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Interim Method for Determining Asbestos in Water

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This manual describes an interim electron microscope (EM) procedure for measuring the concentration of asbestos in water samples. The main features of the method include filtering the sample through a sub-micron polycarbonate membrane filter, examining an EM specimen grid in a transmission electron microscope (TEM), and verifying fiber identity by selected area electron diffraction (SAED).

This interim method is a revision of the procedure issued in 1976 and reflects the improvements that have been made in asbestos analytical methodology since that time.

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INTERIM METHOD FOR DETERMINING ASBESTOS IN WATER

by

Charles H. Anderson and J. MacArthur Long
Analytical Chemistry Branch
Environmental Research Laboratory
Athens, Georgia 30605

ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
ATHENS, GEORGIA 30605

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FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch develops and assesses new techniques for identifying chemical constituents of water and soil.

The widespread use of asbestos-containing materials gives rise to concern about exposure of the general population to low level concentrations in air, water supplies, and food. Although hazards associated with the inhalation of asbestos at high concentrations are recognized, the health significance of ingested particles is not fully understood. An integral part of the U.S. Environmental Protection Agency's research into the health effects of exposure to this naturally occurring mineral is the development of an accurate analysis method for determining its presence in water. To this end, this report presents an interim EPA procedure for analytical laboratories to follow that would result in better agreement of analytical results.

David W. Duttweiler
Director
Environmental Research Laboratory
Athens, Georgia

PREFACE

In July 1976, the Preliminary Interim Method for Determining Asbestos in Water was issued by the U.S. Environmental Protection Agency's Environmental Research Laboratory in Athens, Georgia. That method was perceived as representing the current state-of-the-art in asbestos analytical methodology. The objective of writing the method was to present a procedure that analytical laboratories could follow that would result in a better agreement of analytical results. Since that time, a significant amount of additional experimental work has generated data that provide the basis for a more definitive method than was possible previously.

This revised Interim Method reflects the improvements that have been made in asbestos analytical methodology since the initial procedure was drafted. The general approach to the analytical determination, however, remains the same as previously outlined. That is, asbestos fibers are separated from water by filtration on a sub-micron pore size membrane filter. The asbestos fibers are then counted, after dissolving the filter material, by direct observation in a transmission electron microscope.

The major change in the initial procedure is the elimination of the condensation washer as a means of sample preparation. Intra- and inter-laboratory precision data for the method are presented. Also, a suggested statistical evaluation of grid fiber counts is included.

ABSTRACT

An interim electron microscope (EM) procedure for measuring the concentration of asbestos in water samples is described. The main features of the method include filtering the samples through a sub-micron polycarbonate membrane filter, examining an EM specimen grid in a transmission electron microscope (TEM), and verifying fiber identity by selected area electron diffraction.

This interim method is a revision of the procedure issued in 1976 and reflects the improvements that have been made in asbestos analytical methodology since that time.

This report covers a period from July 1976 to December 1978 and work was completed as of December 1978.

CONTENTS

Foreword iii
Preface iv
Abstract v
Figures and Tables ix

1. Scope and Application 1
2. Summary of Method 1
3. Definitions 2
4. Sample Handling and Preservation 3
   - 4.1 Containment Vessel 3
   - 4.2 Quantity of Sample 3
   - 4.3 Sample Preservation 3
5. Interferences 4
   - 5.1 Misidentification 4
   - 5.2 Obscuration 4
   - 5.3 Contamination 5
   - 5.4 Freezing 5
6. Equipment and Apparatus 5
   - 6.1 Specimen Preparation Laboratory 5
   - 6.2 Instrumentation 6
   - 6.3 Apparatus Supplies and Reagents 7
7. Preparation of Standards 11
   - 7.1 Chrysotile Stock Suspension 11
   - 7.2 Amphibole Stock Suspension 11
   - 7.3 Identification Standards . . 11
8. Procedure 11
   - 8.1 Filtration 11
   - 8.2 Preparation of Electron Microscope Grids . . . 13
   - 8.3 Nuclepore Filter, Modified Jaffe Wick Technique . 14
   - 8.4 Electron Microscopic Examination 16
   - 8.5 Ashing 22
   - 8.6 Determination of Blank Level 23

9. Calculations 24
9.1 Fiber Concentrations 24
9.2 Estimated Mass Concentration 25
9.3 Aspect Ratio 26
10. Reporting 26
11. Precision 26
11.1 Intra-Laboratory 26
11.2 Inter-Laboratory 27
12. Accuracy 27
12.1 Fiber Concentrations 27
12.2 Mass Concentrations 29
13. Suggested Statistical Evaluation of Grid Fiber Counts 29

Bibliography 32

FIGURES

Number		Page
1.	Modified Jaffe Wick Method	8
2.	Illustration of Counting Rules for Field-of-View Method	20

TABLES

Number		Page
1.	Intra-Laboratory Precision	28
2.	Inter-Laboratory Precision	28

INTERIM METHOD
FOR DETERMINING ASBESTOS IN WATER

1. Scope and Application

1.1 This method is applicable to drinking water and water supplies.

1.2 The method determines the number of asbestos fibers per liter, the size (length and width) of the fibers, the size distribution, and the total mass. The method distinguishes chrysotile from amphibole asbestos. The detection limits are variable and depend upon the amount of total extraneous particulate matter in the sample as well as the contamination level in the laboratory environment. Under favorable circumstances, 0.01 million fibers per liter (MFL) can be detected. The detection limit for total mass of asbestos fibers is also variable and depends upon the fiber size and size distribution in addition to the factors affecting the total fiber count. The detection limit under favorable conditions is in the order of 0.1 nanogram per liter (ng/L).

1.3 The method is not intended to furnish a complete characterization of all the fibers in water.

1.4 It is beyond the scope of this method to furnish detailed instruction in electron microscopy, electron diffraction, or crystallography. It is assumed that those using this method will be sufficiently knowledgeable in these fields to understand the methodology involved.

2. Summary of Method

2.1 A variable, known volume of water sample is filtered through a 0.1 micrometer (μm) Nuclepore filter to trap asbestos fibers and the filter is then carbon coated. A small portion of the carbon coated filter with deposited fibers is placed on an electron microscope grid and the filter material is removed by gentle solution in organic solvent. The

material remaining on the electron microscope grid is examined in a transmission microscope at a magnification of about 20,000X. The asbestos fibers are identified by their morphology and electron diffraction patterns and their lengths and widths are measured. The total area examined in the electron microscope is determined and the number of asbestos fibers in this area is counted. The concentration in MFL is calculated from the number of fibers counted, the amount of water filtered, and the ratio of the total filtered area/ sampled filter area. The mass per liter is calculated from the assumed density and the volume of the fibers.

3. Definitions

Asbestos - A generic term applied to a variety of commercially useful fibrous silicate minerals of the serpentine or amphibole mineral groups.

Fiber - Any particulate that has parallel sides and a length/width ratio greater than or equal to 3:1.

Aspect Ratio - The ratio of length to width.

Chrysotile - A nearly pure hydrated magnesium silicate, the fibrous form of the mineral serpentine, possessing a unique layered structure in which the layers are wrapped in a helical cylindrical manner about the fiber axis.

Amphibole Asbestos - A double chain fibrous silicate mineral consisting of Si_4O_{11}, laterally linked by various cations such as aluminum, calcium, iron, magnesium, and sodium. Amphibole asbestos consists of crocidolite and amosite (the fibrous form of cummingtonite-gruenerite), and the fibrous forms of tremolite, actinolite, and anthophyllite. These minerals consist of or contain fibers formed through natural growth processes. Mineral fragments that conform to the definition of a fiber and that are formed through a crushing and milling process are analytically indistinguishable from the naturally formed fibers by this method.

Detection Limit - The calculated concentration in MFL, equivalent to one fiber above the background or blank count (Section 8.6).

Statistically Significant - Any concentration based upon a total fiber count of 5 or more in 20 grid squares.

4. Sample Handling and Preservation

It is beyond the scope of this procedure to furnish detailed instructions for field sampling; the general principles of obtaining water samples apply. Some specific considerations apply to asbestos fibers, however, because they are a special type of particulate matter. These fibers are small, and in water range in length from 0.1 μm to 20 μm or more. Because of the range of size there may be a vertical distribution of particle sizes. This distribution will vary with depth depending upon the vertical distribution of temperature as well as local meteorological conditions. Sampling should take place according to the objective of the analysis. If a representative sample of a water supply is required, a carefully designated set of samples should be taken representing the vertical as well as the horizontal distribution and these samples should be composited for analysis.

4.1 Containment Vessel

The sampling container shall be a clean, screw-capped, polyethylene bottle capable of holding at least 1 liter. The bottle should be rinsed at least two times with the water that is being sampled prior to sampling.

4.2 Quantity of Sample

A minimum of approximately 1 liter of water is required. Leave air space at the top of the container to allow for shaking the sample. It is desirable to obtain two samples from one location.

4.3 Sample Preservation

No preservatives should be added during sampling and the addition of acids should be particularly avoided. If the sample cannot be filtered in the laboratory within 48 hours of its arrival, sufficient amounts (1 milliliter per liter of sample) of a 2.71% solution of mercuric chloride to give a final concentration of 20 ppm of Hg may be added to prevent bacterial growth.

NOTE 1: It has been reported that the growth of algae in water samples can be prevented by storing the samples in the dark.

NOTE 2: Refrigeration of samples at about 5°C minimize bacterial and algal growth.

5. Interferences

5.1 Misidentification

The guidelines set forth in this method for counting fibrous asbestos require a positive identification by both morphology and crystal structure as shown by an electron diffraction pattern. Chrysotile asbestos has a unique tubular structure, usually showing the presence of a central canal, and exhibits a unique characteristic electron diffraction pattern. Although halloysite fibers may show a streaking similar to chrysotile, they do not exhibit chrysotile's characteristic triple set of double spots or 5.3A layer line. It is highly improbable that a non-asbestiform fiber would exhibit the distinguishing chrysotile features. Although amphibole fibers exhibit characteristic morphology and electron diffraction patterns, they do not have the unique properties exhibited by chrysotile. It is possible, therefore, though not probable for misidentification to take place.

It is important to recognize that a significant variable fraction of both chrysotile and amphibole asbestos fibers do not exhibit the required confirmatory electron diffraction pattern. This absence of diffraction is attributable to unfavorable fiber orientation and fiber sizes. The results reported will be low, therefore, as compared to the absolute number of asbestos fibers that are present.

5.2 Obscuration

If large amounts of other materials are present, some small asbestos fibers may not be observed because of physical overlapping. This will result in low values for the reported asbestos content.

5.3 Contamination

Although contamination is not strictly considered to be an interference, it is an important source of erroneous results, particularly for chrysotile. The possibility of contamination, therefore, should always be a consideration.

5.4 Freezing

The effect of freezing on asbestos fibers is not known but there is reason to suspect that fiber breakdown could occur and result in a higher fiber count than was present in the original sample. Therefore, the sample should be transported to the laboratory under conditions that would avoid freezing.

6. Equipment and Apparatus

6.1 Specimen Preparation Laboratory

The ubiquitous nature of asbestos, especially chrysotile, demands that all sample preparation steps be carried out to prevent the contamination of the sample by airborne or other source of asbestos. The prime requirement of the sample preparation laboratory is that it be sufficiently free from asbestos contamination that a specimen blank determination using 200 ml of asbestos-free water yields no more than 2 fibers in 20 grid squares of a conventional 200 mesh electron microscope grid.

In order to achieve this low level of contamination, the sample preparation area should be a separate conventional clean room facility. The room should be operated under positive pressure and have incorporated electrostatic precipitators in the air supply to the room, or as an alternative, absolute (HEPA) filters. No asbestos floor or ceiling tiles, transite heat-resistant boards, or asbestos insulation should be used in construction. Work surfaces should be stainless steel or Formica or equivalent. A laminar flow hood should be provided for sample manipulation. Disposable plastic laboratory coats and disposable overshoes are recommended. Alternatively, new shoes for all operators should be provided and retained for clean room use only. A mat (Tacky Mat, Liberty Industries, 589 Deming Road, Berlin, Connecticut 06037, or equivalent) should be placed at the

entrance to the room to trap any gross contamination inadvertently brought into the room on contaminated shoes. Normal electrical and water services, including a distilled water supply should be provided. In addition, a source of ultra-pure water from a still or filtration-ion exchange system is desirable.

6.2 Instrumentation

6.2.1 Transmission Electron Microscope. A transmission electron microscope that operates at a minimum of 80 kV and has a resolution of better than 1.0 nm and a magnification range of 300 to 100,000 is required. If the upper limit is not attainable directly it may be attained through the use of auxiliary optical viewing. It is mandatory that the instrument be capable of carrying out selected area electron diffraction (SAED) on an area of about 0.3 μm^2. The viewing screen shall have either a millimeter scale, concentric circles of known radii, or other devices to measure the length and width of the fiber. Most modern transmission microscopes meet the requirements for magnification and resolution.

An energy-dispersive X-ray spectrometer is useful for the identification of suspected asbestiform minerals; this accessory to the microscope, however, is not mandatory.

6.2.2 Data Processor. The large number of repetitive calculations make it convenient to use computer facilities together with relatively simple computer programs.

6.2.3 Vacuum Evaporator. A vacuum evaporator is required for depositing a layer of carbon on the Nuclepore filter and for preparing carbon coated grids.

6.2.4 Low Temperature Plasma Asher. An asher is used for the removal of organic material (including the filter) from samples containing so much organic matter that asbestos fibers are obscured. The sample chamber should be at least 10 cm in diameter.

6.3 Apparatus, Supplies and Reagents

6.3.1 Jaffe Wick Washer. The Jaffe Wick Washer for dissolving Nuclepore filter is described in 8.3.1, and is illustrated in Figure 1.

6.3.2 Filtering Apparatus. A 47-mm funnel (Cat No. XX1504700, Millipore Corporation, Order Service Dept., Bedford, MA 01730) or equivalent is used to filter water samples. A 25-mm funnel (Millipore Cat No. XX1002500) or equivalent is used to filter dispersed ash samples.

6.3.3 Vacuum Pump. A pump, for use in sample filtration, should provide vacuum up to about 500 mm of mercury.

6.3.4 EM Grids. Grids of 200-mesh copper or nickel covered with formvar film for use with the Nuclepore-Jaffe sample preparation method are required. These grids may be purchased from manufacturers of electron microscopic supplies or prepared by standard electron microscopic grid preparation procedures. Finder grids may be substituted and are useful if the re-examination of a specific grid opening is desired.

6.3.5 Membrane Filters.

47-mm diameter Millipore membrane filter, type HA, 0.45-μm pore size. Used as a Nuclepore filter support on top of glass frit.

47-mm diameter Nuclepore membrane filter; 0.1-μm pore size (Nuclepore Corp., 7035 Commerce Circle, Pleasanton, CA 94566). Used to filter the water sample.

25-mm diameter Millipore membrane filter, type HA; 0.45-μm pore size. Used as Nuclepore filter support on top of glass frit.

25-mm diameter Nuclepore membrane filter; 0.1-μm pore size. Used to filter dispersed ashed Nuclepore filter.

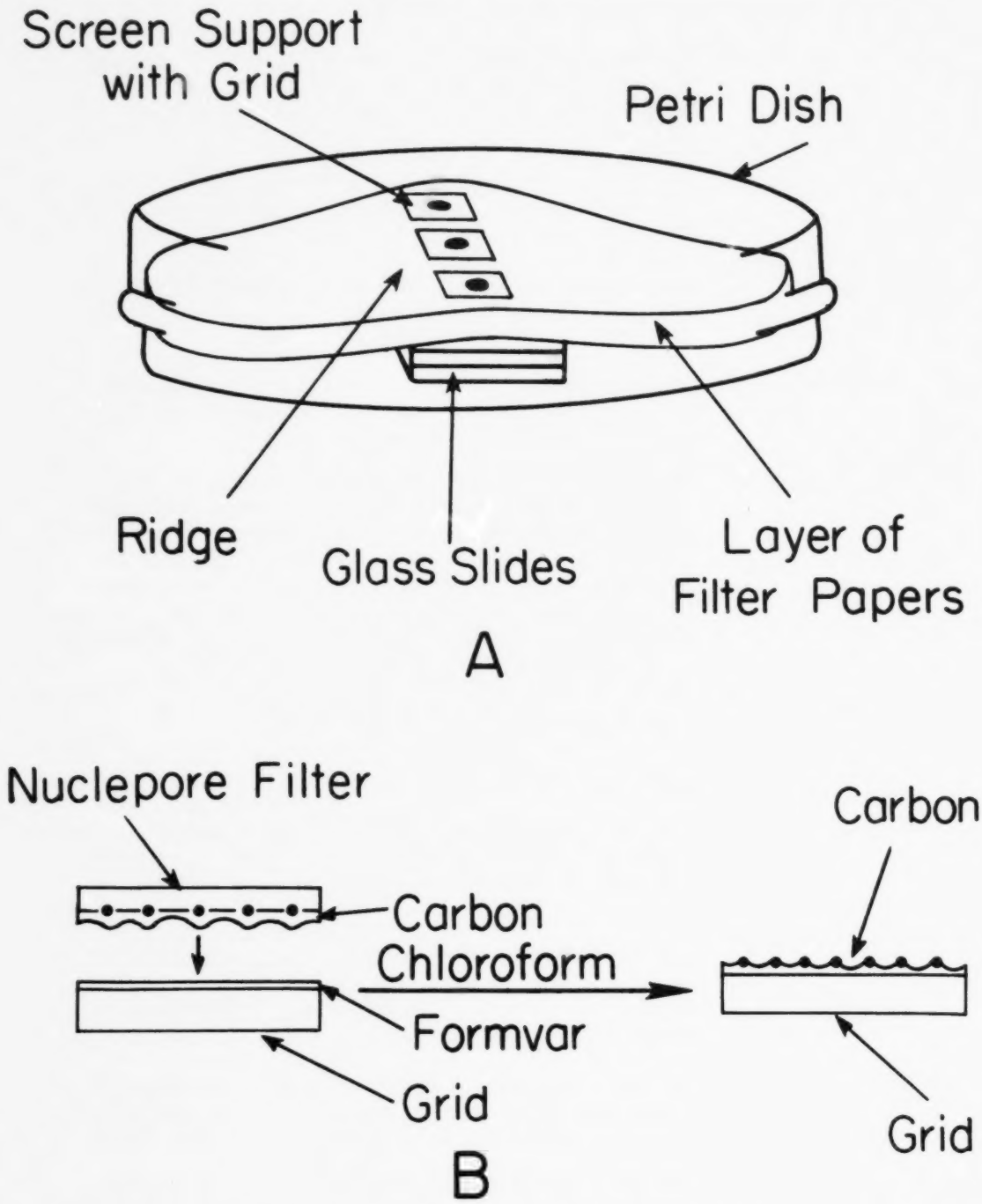

Figure 1. Modified Jaffe Wick Method.
A. Washing Apparatus
B. Washing Process

6.3.6 Glass Vials. 30-mm diameter x 80-mm long. Used to hold filter during ashing.

6.3.7 Glass Slides. Used to support Nuclepore filter during carbon evaporation.

6.3.8 Scalpels.

6.3.9 Scissors.

6.3.10 Tweezers. Several pairs are needed for the many handling operations.

6.3.11 Double-sided Tape. Used to hold filter section flat on glass slide while carbon coating is applied.

6.3.12 Disposable Petri Dishes, 50-mm diameter. Used for storing membrane filters.

6.3.13 Static Eliminator, 500 microcuries Po-210. (Nuclepore Cat. No. V090POL00101) or equivalent. Used to eliminate static charges from membrane filters.

6.3.14 Carbon Rods. Spectrochemically pure, 1/8 in. dia., 3.6 mm x 1.0 mm neck. Used for carbon coating.

6.3.15 Carbon rod sharpener. (Cat. No. 1204, Ernest F. Fullam, Inc., P. O. Box 444, Schenectady, NY 12301) or equivalent. Used for sharpening carbon rods to a neck of specified length and diameter.

6.3.16 Ultrasonic Bath. (50 watts, 55 kHz). Used for dispersing ashed sample and for general cleaning.

6.3.17 Graduated Cylinder, 500 ml.

6.3.18 Spot Plate.

6.3.19 10-µl Microsyringe. Used for administering drop of solvent to filter section during sample preparation.

6.3.20 Carbon Grating Replica, 2160 lines/mm. Used for calibration of EM magnification.

6.3.21 Filter Paper. S & S #589 Black Ribbon (9-cm circles) or equivalent absorbent filter paper. Used for preparing Jaffe Wick Washer.

6.3.22 Screen supports (copper or stainless steel) 12 mm x 12 mm, 200 mesh or equivalent. Used to support specimen grid in Jaffe Wick Washer.

6.3.23 Chloroform. Spectro grade, doubly distilled. Used for dissolving Nuclepore filters.

6.3.24 Asbestos. Chrysotile (Canadian), Crocidolite, Amosite. UICC (Union Internationale Contre le Cancer) Standards. Available from Duke Standards Company, 445 Sherman Avenue, Palo Alto, CA 94306.

6.3.25 Petri Dish. Glass dish (100 mm diameter x 15 mm high). Used for modified Jaffe Wick Washer.

6.3.26 Alconox. (Alconox, Inc., New York, NY 10003) or equivalent. Used for cleaning glassware. Add 7.5 g Alconox to a liter of distilled water.

6.3.27 Parafilm. (American Can Company, Neenah, WI) or equivalent. Used as protective covering for clean glassware.

6.3.28 Pipets. Disposable, 5 ml and 50 ml pipets are required.

6.3.29 Distilled or Deionized Water. Filter if necessary through 0.1-μm Nuclepore filter for making up all reagents, for final rinsing of glassware, and for preparing blanks.

6.3.30 Mercuric chloride, 2.71% solution w/v. Used as sample preservative. See 4.3. Add 5.42 g of reagent grade mercuric chloride ($HgCl_2$) to 100 ml distilled water and dissolve by shaking. Dilute to 200 ml with additional water. Filter through 0.1-μm Nuclepore filter paper before using.

7. Preparation of Standards

Reference standard samples of asbestos that can be used for quality control for a quantitative analytical method are not available. It is, however, necessary for each laboratory to prepare at least two suspensions, one of chrysotile and another of a representative amphibole. These suspensions can then be used for intra-laboratory control and to furnish standard morphology photographs and diffraction patterns.

7.1 Chrysotile Stock Suspension.

Grind about 0.1 g of UICC chrysotile to a powder in an agate mortar. Transfer 10 mg to a clean 1-liter volumetric flask, add several hundred ml of filtered distilled water containing 1 ml of a stock mercuric chloride solution and then make up to 1 liter with filtered distilled water. To prepare a working solution, transfer 10 ml of the above suspension to another 1-liter flask, add 1 ml of a stock mercuric chloride solution and make up to 1 liter with filtered distilled water. This suspension contains 100 μg per liter. Finally transfer 1 ml of this suspension to a 1-liter flask, add 1 ml of a stock mercuric chloride solution and make up to volume with filtered distilled water. The final suspension will contain 5 to 10 MFL and is suitable for laboratory testing.

7.2 Amphibole Stock Suspension.

Prepare amphibole suspensions from UICC amphibole samples as in Section 7.1.

7.3 Identification Standards.

Prepare electron microscopic grids containing the UICC asbestos fibers according to Section 8 and obtain representative photographs of each fiber type and its diffraction pattern for future reference.

8. Procedure

8.1 Filtration.

The separation of insoluble material, including asbestiform minerals, through filtration and subsequent deposition on a membrane filter is a critical step in the procedure. The objective of the

filtration is not only to separate, but also to distribute the particulate matter uniformly such that discrete particles are deposited with a minimum of overlap.

The volume filtered will range from 50 to 500 ml. In an unknown sample, the volume can not be specified in advance because of the presence of variable amounts of particulate matter. In general, sufficient sample is filtered such that a very faint stain can be observed on the filter medium. The maximum loading that can be tolerated is 20 $\mu g/cm^2$, or about 200 μg on a 47-mm diameter filter; 5 $\mu g/cm^2$ is near optimum. If the total solids content is known, an estimate of the maximum volume tolerable can be obtained. In a sample of high solids content, where less than 50 ml is required, the sample should be diluted with filtered distilled water so that a minimum total of 50 ml of water is filtered. This step is necessary to allow the insoluble material to deposit uniformly on the filter. The filtration funnel assembly must be scrupulously cleaned before each filtration. The filtration should be carried out in a laminar flow hood.

NOTE: The following cleaning procedure has been found to be satisfactory.

Wash each piece of glassware three times with distilled water. Following manufacturer's recommendations, use the ultrasonic bath with an Alconox-water solution to clean all glassware. After the ultrasonic cleaning, rinse each piece of glassware three times with distilled water. Then rinse each piece three times with deionized water that has been filtered through 0.1-μm Nuclepore filter. Dry in an asbestos-free oven. After the glassware is dry, seal openings with parafilm.

a. Assemble the vacuum filtration apparatus incorporating the 0.1-μm Nuclepore backed with 0.45-μm Millipore filter. See 8.3.2.

b. Vigorously agitate the water sample in its container. Treatment of the sample in an ultrasonic bath may be required to evenly disperse the particulate material.

c. If the required filtration volume can be estimated, either from turbidity estimates of suspended solids or previous experience, immediately withdraw the proper volume from the container and add the entire volume to the 47-mm diameter funnel. Apply vacuum sufficient for filtration but gentle enough to avoid the formation of a vortex. If a completely unknown sample is being analyzed, a slightly modified procedure must be followed. Pour 500 ml of a well-mixed sample into a 500-ml graduated cylinder and immediately transfer the entire contents to the prepared vacuum filtration apparatus. Apply vacuum gently and continue suction until all of the water has passed through the filter. If the resulting filter appears obviously coated or discolored, another filter should be prepared in the same manner, but this time using only 200 or 100 ml of sample.

NOTE 1: Do not add more water after filtration has started and do not rinse the sides of the funnel.

NOTE 2: Nuclepore filter is basically a hydrophobic material. The manufacturer applies a detergent to the surface of the filter in order to render it hydrophilic; this process, however, does not appear to be entirely satisfactory in some batches. Pretreatment of the filter in a low temperature asher at 10 watts for 10 seconds can be used to render the surface of the filter hydrophilic. This process will significantly decrease the islands of sparse deposit that are frequently observed.

d. Disassemble the funnel, remove the filter, and dry it in a covered petri dish.

8.2 Preparation of Electron Microscope Grids.

Preparation of the grid for examination in the microscope is a critical step in the analytical procedure. The objective is to remove the organic filter material from the asbestos fibers with minimum loss and movement and with minimum breakage of the grid support film.

If the sample contains large amounts of organic matter that interfere with fiber counting and identification a preliminary ashing step is required. See 8.5.

8.3 Nuclepore Filter, Modified Jaffe Wick Technique.

8.3.1 Preparation of Modified Jaffe Washer

Place three glass microscope slides (75 mm x 22 mm) one on top of the other in a petri dish (100 mm x 15 mm) along a diameter. Place 14 S & S #589 Black Ribbon filter papers (9-cm circles) in the petri dish over the stack of microscope slides. Place three copper mesh screen supports (12 mm x 12 mm) along the ridge formed by the stack of slides underneath the layer of filter papers. Place an EM specimen grid on each of the screen supports. See Fig. 1.

NOTE: A stack of 30 to 40 S & S filters (7-cm circle), or equivalent, can be substituted for the 14 filters and microscope slides in preparing the Jaffe washer.

8.3.2 Vacuum Filtration Unit

Assemble the vacuum filtration unit. Place a 0.45-µm Millipore filter type HA on the glass frit and then position a 0.1-µm Nuclepore filter, shiny side up, on top of the Millipore filter. Apply suction to center the filters flat on the frit. Attach the filter funnel and shut off the suction.

8.3.3 Sample Filtration

See 8.1.

8.3.4 Sample Drying

Remove the filter funnel and place the Nuclepore filter in a loosely covered petri dish to dry. The petri dish containing the filter may be placed in an asbestos-free oven at 45° C for 30 minutes to shorten the drying time.

Alternatively, the Nuclepore filter section may be mounted on a glass slide prior to drying the filter.

8.3.5 Selection of Section for Carbon Coating

Using a small pair of scissors or sharp scalpel, cut out a rectangular section of the Nuclepore filter. The minimum approximate dimensions should be 15 mm long and 3 mm wide. Avoid selection near the perimeter of the filtration area.

8.3.6 Carbon Coating the Filter

Tape the two ends of the selected filter section to a glass slide using double-sided tape. Take care not to stretch the filter section. Identify the filter section using a china marker on the slide. Place the glass slide with the filter section into the vacuum evaporator. Insert the necked carbon rod and, following manufacturer's instructions, obtain high vacuum. Evaporate the neck, with the filter section rotating, at a distance of approximately 7.5 cm from the filter section to obtain a 30 to 50 nm layer of carbon on the filter paper. Evaporate the carbon in several short bursts rather than continuously to prevent overheating the surface of the Nuclepore filter.

NOTE 1: Overheating the surface tends to crosslink the plastic, rendering the filter dissolution in chloroform difficult.

NOTE 2: The thickness of the carbon film can be monitored by placing a drop of oil on a porcelain chip that is placed at the same distance from the carbon electrodes as the specimen. Carbon is not visible in the region of the oil drop thereby enabling the visual estimate of the deposit thickness by the contrast differential.

8.3.7 Grid Transfer

Remove the filter from the vacuum evaporator and cut out three sections somewhat less than 3 mm x 3 mm and such that the

square of Nuclepore fits within the circumference of the grid. Pass each of the filter sections over a static eliminator and then place each of the three sections carbon-side down on separate specimen grids previously placed in the modified Jaffe Washer. Using a microsyringe, place a 10-µl drop of chloroform on each filter section resting on a grid and then saturate the filter pad until pooling of the solvent occurs below the ridge formed by the glass slides inserted under the layer of filter papers. Place the cover on the petri dish and allow the grids to remain in the washer for approximately 24 hours. Do not allow the chloroform to completely evaporate before the grids are removed. To remove the grids from the washer, lift the screen support with the grid resting upon it and set this in a spot plate depression to allow evaporation of any solvent adhering to the grid. The grid is now ready for analysis or storage.

8.4 Electron Microscopic Examination

8.4.1 Microscope Alignment and Magnification Calibration

Following the manufacturer's recommendations carry out the necessary alignment procedures for optimum specimen examination in the electron microscope. Calibrate the routinely used magnifications using a carbon grating replica.

NOTE: Screen magnification is not necessarily equivalent to plate magnification.

8.4.2 Grid Preparation Acceptability

After inserting the specimen into the microscope, adjust the magnification low enough (300X-1000X) to permit viewing complete grid squares. Inspect at least 10 grid squares for fiber loading and distribution, debris contamination, and carbon film continuity.

Reject the grid for counting if:

1) The grid is too heavily loaded with fibers to perform accurate counting and diffraction operations. A new sample preparation either from a smaller volume of water or from a dilution with filtered distilled water must then be prepared.

2) The fiber distribution is noticeably uneven. A new sample preparation is required.

3) The debris contamination is too severe to perform accurate counting and diffraction operations. If the debris is largely organic the filter must be ashed and redispersed (see 8.5). If it is inorganic, the sample must be diluted and again prepared.

4) The majority of grid squares examined have broken carbon films. A different grid preparation from the same initial filtration must be substituted.

8.4.3 Procedure for Fiber Counting

Two methods are commonly used for fiber counting. In one method (A), 100 fibers contained in randomly selected fields of view are counted. The number of fields plus the area of a field of view must be known when using this method. In the other method (B), all fibers (at least 100) in several grid squares or 20 grid squares are counted. The number of grid squares counted and the average area of one grid square must be known when using this method.

NOTE: The method to use depends upon the fiber loading on the grid and it is left to the judgment of the analyst to select the optimum method. The following guidelines can be used: If it is estimated that a grid square (80 μm x 80 μm) contains 50 to 100 fibers at a screen magnification of 20000X, it is convenient to use the field-of-view counting method. If the estimate is less than 50, the grid square method of counting should be chosen. On the other

hand, if the fiber count is estimated to be over 300 fibers per grid square, a new grid containing fewer fibers must be prepared (through dilution or filtration of a smaller volume of water).

8.4.3A Field-of-View Method

After determining that a fiber count can be obtained using this method, adjust the screen magnification to 15,000 to 20,000X. Select a number of grid squares that would be as representative as possible of the entire analyzable grid surface. From each of these squares, select a sufficient number of fields of view for fiber counting. The number of fields of view per grid square is dependent upon the fiber loading. If more than one field of view per grid square is selected, scan the grid opening orthogonally in an arbitrary pattern that prevents overlapping of fields of view. Carry out the analysis by counting, measuring and identifying (see 8.4.4) approximately 50 fibers on each of two grids.

The following rules should be followed when using the field of view method of fiber counting. Although these rules were derived for a circular field of view they can be modified to apply to square or rectangular designs.

1) Count all fibers contained within the counting area and not touching the circumference of the circle.

2) Designate the upper right-hand quadrant as I and number in clockwise order. Count all fibers touching or intersecting the arc of quadrants I or IV. Do not count fibers touching or intersecting the arc of quadrants II or III.

3) If a fiber intersects the arc of both quadrants III and IV or I and II count it only if the greater length was outside the arc of quadrants IV and I, respectively.

4) Count fibers intersecting the arc of both quadrants I and III but not those intersecting the arc of both II and IV.

These rules are illustrated in Fig. 2.

8.4.3B Grid Square Method

After determining that a fiber count can be obtained using this method adjust the screen magnification to 15,000 to 20,000X. Position the grid square so that scanning can be started at the left upper corner of the grid square. While carefully examining the grid, scan left to right, parallel to the upper grid bar. When the perimeter of the grid square is reached, adjust the field of view down one field width and scan in the opposite direction. The tilting section of the fluorescent screen may be used conveniently as the field of view. Examine the square until all the area has been covered. The analysis should be carried out by counting, measuring, and identifying (see 8.4.4) approximately 50 fibers on each of two grids or until 10 grid squares on each of two grids have been counted. Do not count fibers intersecting a grid bar.

8.4.4 Measurement and Identification

Measure and record the length and width of each fiber having an aspect ratio greater than or equal to three. Disregard obvious biological-bacteriological fibers and diatom fragments. Examine the morphology of each fiber using optical viewing if necessary. Tentatively identify, by reference to the UICC standards, chrysotile or possible amphibole asbestos. Attempt to obtain a diffraction pattern of each fiber utilizing the shortest camera length possible. Move the suspected fiber image to the center of the screen and insert a suitable selected area aperture into the electron beam so that the fiber image, or a portion of it, is in the illuminated area. The size of the aperture and the portion of the fiber should be such that particles other than the one to be examined are

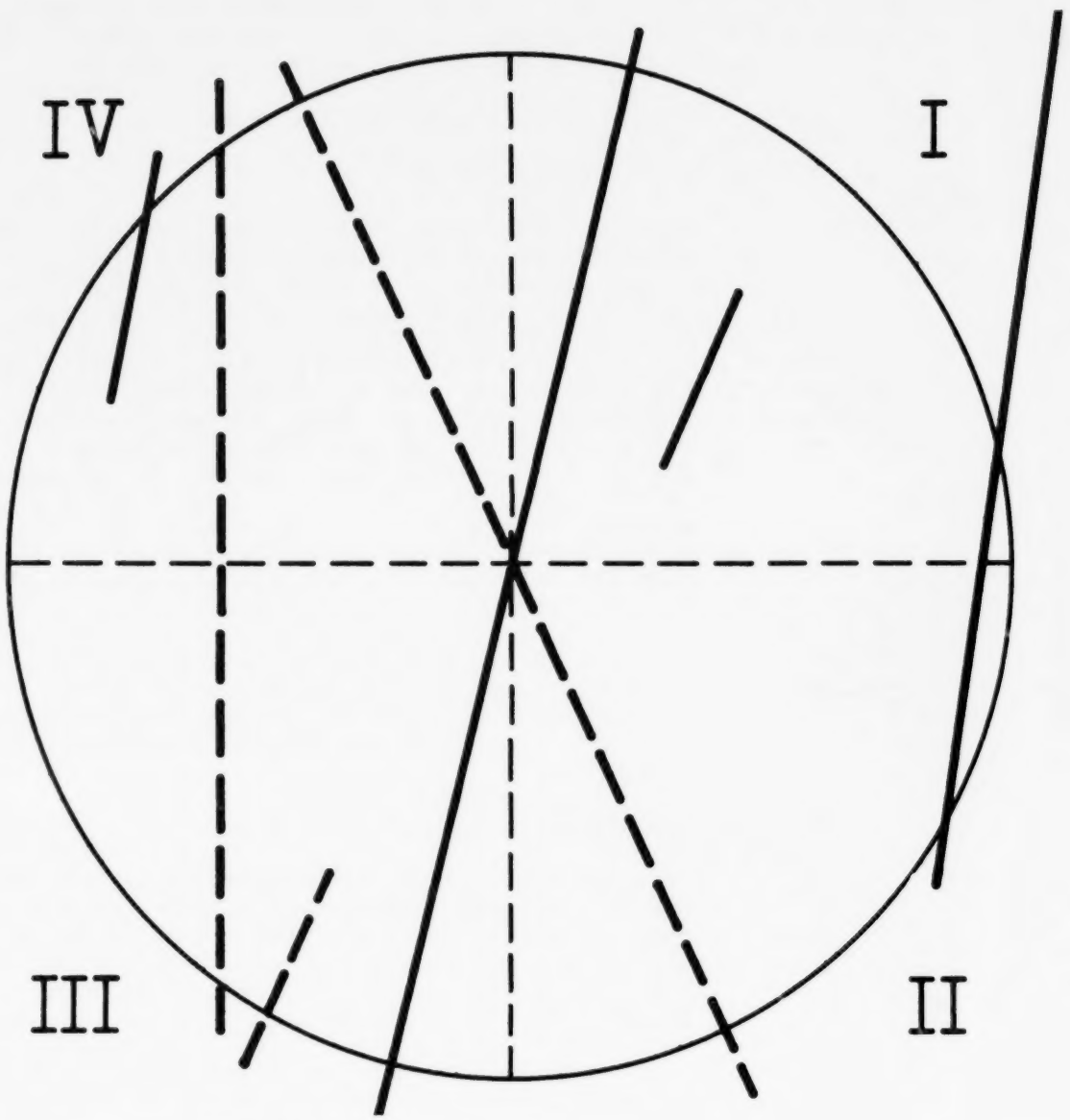

Figure 2. Illustration of Counting Rules for Field of View Method.

excluded from the selected area. Observe the diffraction pattern with the binocular attachment. If an incomplete diffraction pattern is obtained, move the particle image around in the selected area to get a clearer diffraction pattern or to eliminate possible interferences from neighboring particles.

Determine whether the fiber is chrysotile or an amphibole by comparing the diffraction pattern obtained to the diffraction patterns of known standard asbestos fibers. Confirm the tentative identification of chrysotile and amphibole asbestos from their electron diffraction patterns. Classify each fiber as chrysotile, amphibole, non-asbestos, no diffraction, or ambiguous.

NOTE 1: It is convenient to use a tape recorder during the examination of the fibers to record all pertinent data. This information can then be summarized on data sheets or punched cards for subsequent automatic data processing.

NOTE 2: Chrysotile fibers occur as single fibrils or in bundles. The fibrils generally show a tubular structure with a hollow canal, although the absence of the canal does not rule out its identification. Amphibole asbestos fibers usually exhibit a lath-like structure with irregular ends, but occasionally will resemble chrysotile in appearance.

NOTE 3: The positive identification of asbestos by electron diffraction requires some judgment on the part of the analyst because some fibers give only partial patterns. Chrysotile shows unique prominent streaks on the layer lines nearest the central one and a triple set of double spots on the second layer line. The streaks and the set of double spots are the distinguishing characteristics of chrysotile required for identification. Amphibole asbestos requires a more complete diffraction pattern to be positively identified. As a qualititative guideline,

layer lines for amphibole, without the unique streaks (some streaking may be present) of chrysotile, should be present and the arrangement of diffraction spots along the layer lines should be consistent with the amphibole pattern. The pattern should be distinct enough to establish these criteria.

NOTE 4: Chrysotile and thin amphibole fibers may undergo degradation in an electron beam; this is particularly noticeable in small fibers. It may exhibit a pattern for 1 to 2 seconds and disappear and the analyst must be alert to note the characteristic features.

NOTE 5: An ambiguous fiber is a fiber that gives a partial electron diffraction pattern resembling asbestos, but is insufficient to provide positive identification.

8.4.5 Determination of Grid Square Area

Measure the dimensions of several representative grid squares from each batch of grids with an optical microscope. Calculate the average area of a grid square. This should be done to compensate for variability in grid square dimensions.

8.5 Ashing

Some samples contain sufficiently high levels of organic material that an ashing step is required before fiber identification and counting can be carried out.

Place the dried Nuclepore filter paper containing the collected sediment into a glass vial (28 mm diameter x 80 mm high). Position the filter such that the filtration side touches the glass wall. Place the vial in an upright position in the low temperature asher. Operate the asher at 50 watts (13.56 MHz) power and 2 torr oxygen pressure. Ash the filter until a thin film of white ash remains. The time required is generally 6 to 8 hours. Allow the ashing chamber to slowly reach atmospheric pressure and remove the vial. Add 10 ml of filtered distilled water to the vial. Place the vial in an ultrasonic bath for 30 minutes to disperse the ash. Dilute the sample if required.

Assemble the 25-mm diameter filtering apparatus. Center a 25-mm diameter 0.1-μm Nuclepore filter (with the 0.45-μm Millipore backing) on the glass frit. Apply suction and recenter the filter if necessary. Attach the filter funnel and turn off the suction. Add the water containing the dispersed ash from the vial to the filter funnel. Apply suction and filter the sample. After drying, this filter is ready to be used in preparing sample grids as in 8.3.

NOTE 1: In specifying a 25-mm diameter filter it is assumed that the ashing step is necessary mainly because of the presence of organic material and that the smaller filtering area is desirable from the point of view of concentrating the fibers. If the sample contains mostly inorganic debris such that the smaller filtering area will result in overloading the filter, the 47-mm diameter filter should be used.

NOTE 2: It will be noted that a 10-ml volume is filtered in this case instead of the minimum 50-ml volume specified in 8.1. These volumes are consistent when it is considered that there is approximately a 5-fold difference in effective filtration area between the 25-mm diameter and 47-mm diameter filters.

NOTE 3: Cross contamination is probable when ashing more than one sample at a time.

8.6 Determination of Blank Level

Carry out a blank determination with each batch of samples prepared, but a minimum of one per week. Filter a fresh supply (500 ml) of distilled, deionized water through a clean 0.1-μm membrane filter. Filter 200 ml of this water through a 0.1-μm Nuclepore filter, prepare the electron microscope grid, and count exactly as in the procedures 8.1 - 8.4. Examine 20 grid squares and record this number of fibers. A maximum of two fibers in 20 grid squares is acceptable for the blank sample.

NOTE: Monitoring the background level of asbestos is an integral part of the procedure. Upon initiating asbestos analytical work, blank samples must be run to establish the initial suitability of the laboratory environment, cleaning procedures, and

reagents for carrying out asbestos analyses. Analytical determinations of asbestos can be carried out only after an acceptably low level of contamination has been established.

9. Calculations

9.1 Fiber Concentrations

Grid Square Counting Method - If the Grid Square Method of counting is employed, use the following formula to calculate the total asbestos fiber concentration in MFL.

$$C = (\overline{F} \times A_f)/(A_g \times V_o \times 1000) \qquad (1)$$

where: C = Fiber concentration (MFL)

$\overline{F}$ = Average number of fibers per grid opening

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

A_g = Average area of one grid square (mm^2)

V_o = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter. If one-half the filter is ashed, multiple C by two.

Field-of-View Counting Method - If the Field-of-View Method of counting is employed, use the following formula to calculate the total asbestos fiber concentrations (MFL).

$$C = (\overline{F} \times A_f \times 1000)/(A_v \times V_o) \qquad (2)$$

where: C = Fiber concentration

$\overline{F}$ = Average number of fibers per field of view

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

A_v = Area of one field of view (μm^2)

V_o = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter.

9.2 Estimated Mass Concentration

Calculate the mass (μg) of each fiber counted using the following formula.

$$M = L \times W^2 \times D \times 10^{-6}$$

If the fiber content is predominantly chrysotile, the following formula may be used.

$$M = \frac{\pi}{4} \times L \times W^2 \times D \times 10^{-6} \qquad (3)$$

where: M = Mass (μg)

L = Length (μm)

W = Width (μm)

D = Density of fibers (g/cm^3)

Then calculate the mass concentration ($\mu g/l$) employing the following formula.

$$M_c = C \times \bar{M}_f \times 10^6$$

where: M_c = mass concentration ($\mu g/l$)

C = fiber concentration (MFL)

$\bar{M}_f$ = mean mass per fiber (μg)

To calculate $\bar{M}_f$ use the following formula.

$$\bar{M}_f = \sum_{i=1}^{n} M_{i/n} \qquad (4)$$

where: M_i = mass of each fiber, respectively

n = number of fibers counted

NOTE 1: Because many of the amphibole fibers are lath shaped rather than square in cross section the computed mass will tend to be high because laths will, in general, tend to lie flat rather than on edge.

NOTE 2: Assume the following densities: chrysotile 2.5, amphibole 3.25.

9.3 Aspect Ratio

The aspect ratio for each fiber is calculated by dividing the length by the width.

10. Reporting

10.1 Report the following concentration as MFL for sample and blank using 95% confidence intervals.

a. Chrysotile

b. Amphibole

c. Total asbestos fibers

10.2 Use two significant figures for concentrations greater than 1 MFL, and one significant figure for concentrations less than 1 MFL.

10.3 Tabulate the size distribution, length and width.

10.4 Tabulate the aspect ratio distribution.

10.5 Report the calculated mass as µg/l.

10.6 Indicate the detection limit in MFL.

10.7 Indicate if less than five fibers were counted.

10.8 Include remarks concerning pertinent observations, (clumping, amount of organic matter, debris) amount of suspected though not identifiable as asbestos fibers (ambiguous).

11. Precision

11.1 Intra-Laboratory

The precision that is obtained within an individual laboratory is dependent upon the number of fibers counted. If 100 fibers are counted and the loading

is at least 3.5 fibers/grid square, computer modeling of the counting procedure shows that a relative standard deviation of about 10% can be expected.

In actual practice some degradation from this precision will be observed but should not exceed ± 15% if several grids are prepared from the same filtered sample. The relative standard deviation of analyses of the same water sample in the same laboratory will increase as a result of sample preparation errors and a relative standard deviation of about about ± 25 to 35% will occur. As the number of fibers counted decreases, the precision will also decrease approximately proportional to $\sqrt{N}$ where N is the number of fibers counted. The precision for mass concentration is generally poorer than that for fiber concentration.

Based upon the analysis of one laboratory utilizing a different analyst for each of three water samples, intra-laboratory precision data are presented in Table 1.

11.2 Inter-Laboratory

Based upon the analysis by various government and private industry laboratories of filters prepared from nine water samples, inter-laboratory precision data of the method are presented in Table 2.

12. Accuracy

12.1 Fiber Concentrations

As no standard reference materials are available, only approximate estimates of the accuracy of the procedure can be made. At 1 MFL, it is estimated that the results should be within a factor of 10 of the actual asbestos fiber content.

This method requires the positive identification of a fiber to be asbestos as a means for its quantitative determination. As the state-of-the-art precludes the positive identification of all of the asbestos fibers present, the results of this method, as expressed as MFL, will be biased on the low side and, assuming no fiber loss, represent 0.4 to 0.8 of the total asbestos fibers present.

TABLE 1. INTRA-LABORATORY PRECISION

Sample Type	Number of Sample Aliquots Analyzed	Mean Fiber Concentration MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation	Mass Concentration (µg/l)	Precision, Relative Standard Deviation
Chrysotile (UICC)	26	23	37%	0.32	71%
Crocidolite (UICC)	20	8	36%	1.5	48%
Taconite (raw water)	20	16	24%	10.5	65%

TABLE 2. INTER-LABORATORY PRECISION

	Sample Type	Number of Labs Reporting	Mean Fiber Concentration, MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation
	Chrysotile	10	877	35%
		9	119	43%
		11	59	41%
		9	31	65%
		9	28	32%
		3	25	35%
	Amphibole	11	139	50%
		4	95	52%
		14	36	66%

12.2 Mass Concentrations

As in the case of the fiber concentrations, no standard samples of the size distribution found in water are available. The estimated mass concentration is often very inaccurate because of poor counting statistics associated with large fibers that are few in number but represent most of the actual mass concentration.

13. Suggested Statistical Evaluation of Grid Fiber Counts

13.1 Because the fiber distribution on the sample filter, resulting from the method of filtration, has not been fully characterized, the fiber distribution obtained on the electron microscope grids for each sample should be tested statistically against an assumed distribution and a measure of the precision of the analysis should be provided.

13.2 Assume that the fibers are uniformly and randomly distributed on the sample filter and grids. One method for confirming this assumption is given below.

Using the chi-square test, determine whether the total number of fibers found in individual grid openings are randomly and uniformly distributed among the openings using the following formula.

$$X^2 = \sum_{i=1}^{N} \frac{(n_i - np_i)^2}{np_i} \qquad (5)$$

where: X^2 = Chi-square statistic

N = Number of grid openings examined for the sample

n_i = Total number of fibers found in each respective grid opening

n = Total number of fibers found in N grid openings

p_i = Ratio of the area of each respective grid opening to the sum of the areas of all grid openings examined

NOTE: If an average area for the grid squares has been measured as outlined in 8.4.5, the term np_i represents the mean fiber count per grid square.

If the value for X^2 exceeds the value listed in statistical tables for the 0.1% significance level with N-1 degrees of freedom, the fibers are not considered to be uniformly and randomly distributed among the grid openings. In this case, it is advisable to try to improve the uniformity of fiber deposition by filtering another aliquot of the sample and repeating the analysis.

13.3 If uniformity and randomness of fiber deposition on the microscope grids has been demonstrated as in 13.2, and the fiber concentration is assumed to be normally distributed about the mean value, the 95% confidence interval about the mean fiber concentrations for chrysotile, amphibole, and total asbestos fibers may be determined using the following formulae.

$$S_c = \left[\frac{N \sum_{i=1}^{N} X_i^2 - \left(\sum_{i=1}^{N} X_i \right)^2}{N(N-1)} \right]^{1/2} \qquad (6)$$

where: S_c = Standard deviation of the chrysotile fiber count

N = Number of grid openings examined for the sample

X_i = Number of chrysotile fibers in each grid opening, respectively

Obtain the standard deviations of the fiber counts for amphibole asbestos fibers and for total asbestos fibers by substituting the corresponding value of X_i into equation (6).

$$X_u = \bar{X} + \frac{tS_c}{\sqrt{N}} \qquad (7)$$

$$X_L = \bar{X} - \frac{tS_c}{\sqrt{N}} \qquad (8)$$

where: X_u = Upper value of 95% confidence interval for chrysotile

X_L = Lower value of 95% confidence interval for chrysotile

$\overline{X}$ = Average number of fibers per grid opening

t = Value listed in t-distribution tables at the 95% confidence level for a two tailed distribution with N-1 degree of freedom

S_c = Standard deviation of the fiber counts for chrysotile

N = Number of grid openings examined for the sample

The values of X_u and X_L can be converted to concentrations in millions of fibers per liter using the formula in section 9 and substituting either X_u or X_L for the term F.

Obtain the upper and lower values of the 95% confidence interval for amphibole asbestos fibers and total asbestos fibers by substituting the corresponding values of $\overline{X}$ and S into equations (7) and (8).

Report the precision of the analysis, in terms of the upper and lower limits of the 95% confidence interval, for chrysotile, amphibole, and total asbestos fiber content. If a lower limit is found to be negative, report the value of the limit as zero.

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